Ultra-Small-Angle X-ray Scattering on Nano-Particle Monolayers

Hendrik K. Kammler[1,2], Gregory Beaucage[2], Sotiris E. Pratsinis[1]
[1] Particle Technology Laboratory, ETH Zürich, CH-8092 Zürich, Switzerland; http://www.ptl.ethz.ch
[2] Department of Material Science and Engineering, University of Cincinnati, Cincinnati, OH-45221-0012, USA

Introduction

Monolayers of silica nanoparticles were collected on metal substrate from a flame aerosol reactor by **thermophoretic sampling (TS)**. The third generation synchrotron source coupled with ultra-small-angle x-ray scattering (USAXS) camera at APS (UNICAT) was capable of measuring a reasonable scattering pattern on such thermophoretically collected samples. This new **TS-USAXS technique** enables monolayer-like particle deposition. It further allows the collection of localized samples for USAXS analysis, thus, enabling point wise mapping of flame reactors. Data along certain streamlines or 3-D mapping is possible.

Local particle samples with extremely **narrow size distributions** were found indicating a much higher degree of uniformity in particle size than previously expected from powders samples on similar aerosol streams.

Growth of Nanoparticles

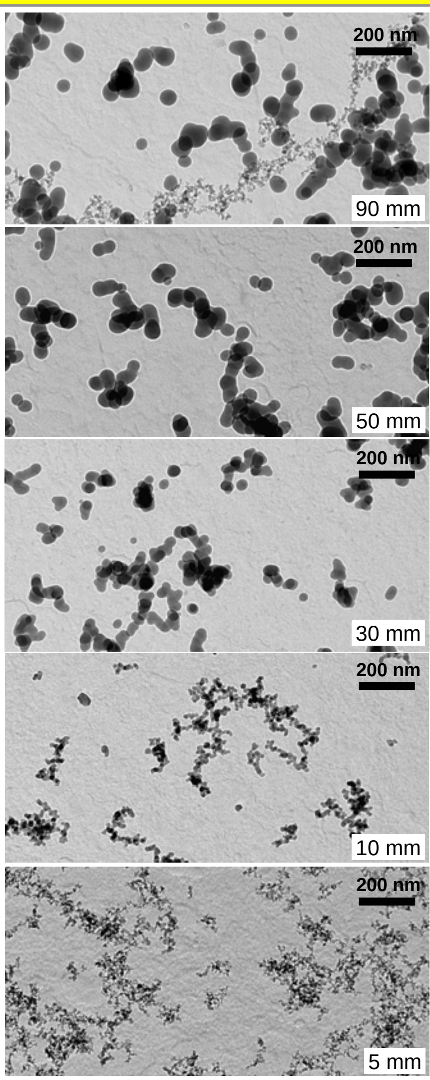


*Fig. 1: Silica particles as collected by conventional thermophoretic sampling (TS) [1,2]. The growth of particles can be visualized. Using aluminum foil in-stead of TEM grids and performing multiple sampling from the same location in the flame, the Al-probe was covered with **a silica monolayer** [1] (as indicated in Fig. 2).*

Monolayer Deposition

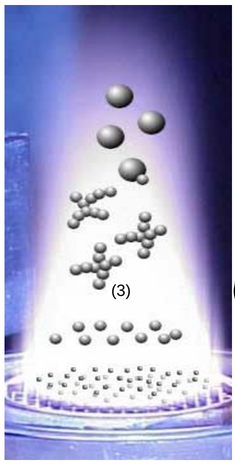
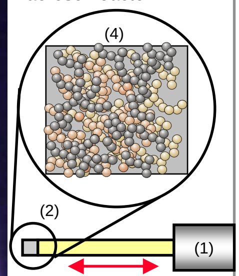


Fig. 2: Premixed flame aerosol reactor.



A pneumatic piston (1) drives the probe (2) three times to a well defined location in the flame (3) and particles deposite by thermophoresis to collect the monolayer (4) [1,2].

Scattering of Monolayers

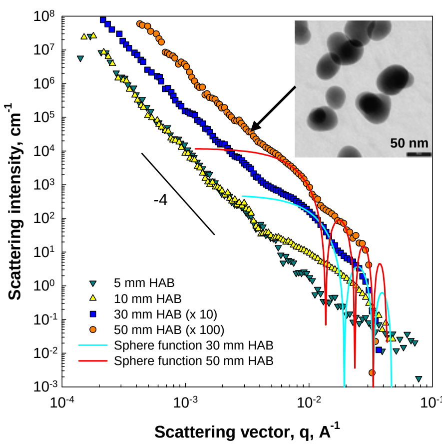


*Fig. 3: At 5 mm height above burner (HAB), scattering from small particles is detected. At 10 mm HAB, which corresponds to 2 ms of particle residence time in the flame, grown particles are detected. With increasing HAB, particles grow even larger into spheres with **low polydispersity**. Spectra at 30 and 50 mm HAB can be described well with the **sphere function** (solid lines)[3]:*

$$I(q) = A\cdot\left(3\cdot\frac{\sin(q\cdot R)-(q\cdot R)\cdot\cos(q\cdot R)}{(q\cdot R)^3}\right)^2$$

The slope of -4 at low q values is probably from the Al-foil used for particle deposition. The same samples were measured as well at ESRF in Grenoble, France, and showed identical scattering patterns.

References

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Monitoring growth dynamics

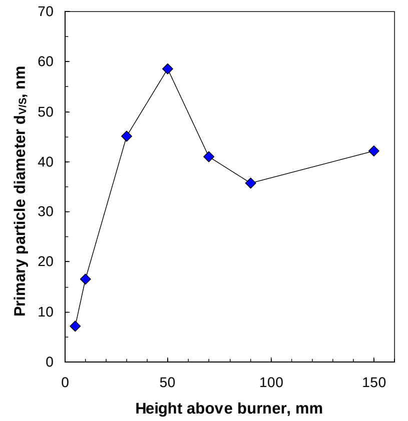

Fig. 4: The primary particle size, $d_{V/S}$, obtained by USAXS evaluation [4,5] increases up to 50 mm height above the burner (HAB) as particles collide and coagulate/coalesce at high temperature. At the flame edge, small particles are formed since temperature is significantly lower [2]. Above 50 mm HAB, flame turbulence brings large agglomerates with these small primary particles to the flame center, decreasing the average primary particle diameter [2], as observed here by TS-USAXS.

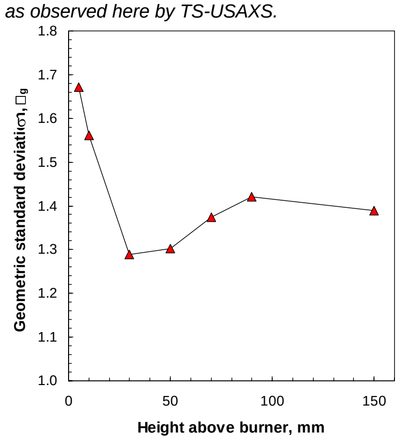

Fig. 5: σ_g, goes through a minimum indicating narrow size distributiuions of $\sigma_g < 1.3$ (at 30 and 50 mm HAB), which is well below the self-preserving limit of 1.45 for coagulation. This corresponds well with Fig. 4.

Conclusions

- *Good scattering patterns were obtained from silica nanoparticle monolayers by ultra-small-angle x-ray scattering.*
- *Along particle formation, regions of low polydispersity were idintifird by this new scampling technique. The sphere function described these narrow scattering pattern with high accuracy*

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